

**PATENT**

Attorney Docket No.: AMAT/5190/CPI/COPPER/PJS  
Express Mail No.: EV351032116US

**Claims:**

1. An apparatus for controlling delivery of a precursor from a vessel to a process chamber, comprising:

    a first valve adapted to regulate a first carrier gas flowing into the vessel, whereby the first carrier gas is combined with the precursor;

    an output from the vessel in fluid communication with the process chamber;  
    a second valve adapted to regulate a second carrier gas flowing to the process chamber whereby the first and second carrier gases and the precursor are combined to form a process gas;

    a gas analyzer having an ultrasonic transducer or a mass flow meter adapted to generate a signal indicative of a concentration of the precursor in the process gas; and

    a controller configured to calculate a mass flow rate of the precursor based on the signal.

2. The apparatus of claim 1, wherein the controller is configured to adjust both the first valve and the second valve while the precursor is pulsed into the process chamber at a period of time in a range from about 0.01 second to about 5 seconds.

3. The apparatus of claim 1, wherein the controller is configured to adjust both the first valve and the second valve while maintaining the process gas with a constant flow.

4. The apparatus of claim 1, wherein the controller is configured to adjust the temperature of the vessel to change the concentration of the precursor in the process gas.

5. The apparatus of claim 4, wherein the controller is configured to adjust the first valve and the second valve while maintaining the process gas with a constant flow.

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6. The apparatus of claim 1, wherein the first valve adjusts to increase or decrease the concentration of the precursor in the process gas.
7. The apparatus of claim 6, wherein the second valve adjusts to maintain a constant flow.
8. The apparatus of claim 1, wherein the first carrier gas and the second carrier gas are the same.
9. The apparatus of claim 1, wherein the first carrier gas and the second carrier gas are selected from the group consisting of argon, nitrogen, helium, hydrogen and combinations thereof.
10. The apparatus of claim 9, wherein the precursor is selected from the group consisting of  $W(CO)_6$ ,  $(Me_2N)_5Ta$ ,  $(Et_2N)_5Ta$ ,  $(^tBuN)Ta(NMe_2)_3$ ,  $(^tBuN)Ta(NEt_2)_3$ ,  $TaCl_5$ ,  $TaF_5$ ,  $TiCl_4$ ,  $HfCl_4$ ,  $(Et_2N)_4Hf$  and  $XeF_2$ .
11. A system comprising:
  - a process chamber;
  - a gas delivery system adapted to deliver a precursor from a vessel containing the precursor to the process chamber via a process gas produced by flowing a first carrier gas into the vessel and combining the first carrier gas with a second carrier gas flowing through a bypass around the vessel;
  - a precursor monitoring apparatus disposed between the process chamber and the vessel, wherein the precursor monitoring apparatus has a gas analyzer to generate a signal indicative of a concentration of the precursor in the process gas or the signal is indicative of the flow rate of the precursor; and
  - an integral controller to receive the signal.

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12. The apparatus of claim 11, wherein the integral controller is configured to adjust both the process gas to be pulsed into the process chamber at a period of time in a range from about 0.01 second to about 5 seconds.
13. The system of claim 11, wherein a first valve controls the first carrier gas and a second valve controls the second carrier gas.
14. The system of claim 13, wherein the integral controller is configured to adjust both the first valve and the second valve while maintaining the process gas with a constant flow.
15. The system of claim 11, wherein the integral controller is configured to increase a temperature of the vessel to increase the concentration of the precursor.
16. The system of claim 15, wherein the integral controller is configured to adjust the first valve and the second valve while maintaining the process gas with a constant flow.
17. The system of claim 13, wherein the first valve is adjusted to increase or decrease the concentration of the precursor in the process gas.
18. The system of claim 17, wherein the second valve is adjusted to maintain a constant flow.
19. The system of claim 11, wherein the first carrier gas and the second carrier gas are the same.
20. The system of claim 11, wherein the gas analyzer is selected from the group consisting of ultrasonic transducers, infrared spectroscopy, ultraviolet spectroscopy, gas chromatography, mass spectroscopy, mass flow meters and combinations thereof.

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21. The system of claim 20, wherein the first carrier gas and the second carrier gas are selected from the group consisting of argon, nitrogen, helium and hydrogen.
22. The system of claim 21, wherein the precursor is selected from the group consisting of  $W(CO)_6$ ,  $(Me_2N)_5Ta$ ,  $(Et_2N)_5Ta$ ,  $(^tBuN)Ta(NMe_2)_3$ ,  $(^tBuN)Ta(NEt_2)_3$ ,  $TaCl_5$ ,  $TaF_5$ ,  $TiCl_4$ ,  $HfCl_4$ ,  $(Et_2N)_4Hf$  and  $XeF_2$ .
23. An apparatus for delivering of a precursor from a vessel to a process chamber, comprising:
  - a first valve to regulate a first carrier gas flowing through an input into the vessel;
  - an output from the vessel in fluid communication with the process chamber;
  - a second valve to regulate a second carrier gas flowing to the process chamber;
  - a process gas comprising the first carrier gas, the second carrier gas and the precursor;
  - a gas analyzer to generate a signal indicative of a concentration of the precursor in the process gas or indicative of a process flow rate; and
  - a controller to receive the signal and is configured to maintain the concentration of the precursor and the volume flow rate of the process gas constant by adjusting the first valve and the second valve.
24. The method of claim 23, wherein the precursor is pulsed into the process chamber at a period of time in a range from about 0.01 second to about 5 seconds.
25. The system of claim 23, wherein the controller is configured to increase a temperature of the vessel to increase the concentration of the precursor.
26. The system of claim 23, wherein the first valve adjusts to increase or decrease the concentration of the precursor in the process gas.

27. The system of claim 26, wherein the second valve adjusts to maintain the process gas constant.
28. The system of claim 23, wherein the first carrier gas and the second carrier gas are the same.
29. The system of claim 23, wherein a gas analyzer is selected from the group consisting of ultrasonic transducers, infrared spectroscopy, ultraviolet spectroscopy, gas chromatography, mass spectroscopy, mass flow meter and combinations thereof.
30. The system of claim 29, wherein the first carrier gas and the second carrier gas are selected from the group consisting of argon, nitrogen, helium and hydrogen.
31. The system of claim 30, wherein the precursor is selected from the group consisting of  $W(CO)_6$ ,  $(Me_2N)_5Ta$ ,  $(Et_2N)_5Ta$ ,  $(^tBuN)Ta(NMe_2)_3$ ,  $(^tBuN)Ta(NEt_2)_3$ ,  $TaCl_5$ ,  $TaF_5$ ,  $TiCl_4$ ,  $HfCl_4$ ,  $(Et_2N)_4Hf$  and  $XeF_2$ .
32. A method for monitoring and controlling delivery of a precursor from a vessel to a process chamber, comprising:
  - measuring a concentration of the precursor in a process gas, wherein the process gas is produced by flowing a first carrier gas through the vessel and then combining with a second carrier gas; and
  - calculating a mass flow rate of the precursor based on the measured concentration of the precursor in the process gas, the first carrier gas flow and the second carrier gas flow.
33. The method of claim 32, wherein the precursor is pulsed into the process chamber at a period of time in a range from about 0.01 second to about 5 seconds.

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34. The method of claim 32, wherein regulating the first carrier gas and the second carrier gas creates a constant flow for the process gas.
35. The method of claim 32, wherein the first carrier gas is controlled by a first valve and the second carrier gas is controlled by a second valve.
36. The method of claim 35, wherein an integral controller is configured to adjust both the first valve and the second valve while maintaining the process gas with a constant flow.
37. The method of claim 36, wherein increasing a temperature of the vessel via the integral controller increases the concentration of the precursor.
38. The method of claim 37, wherein adjusting the first valve and the second valve via the integral controller maintains the process gas with a constant flow.
39. The method of claim 32, wherein measuring the concentration of the precursor uses a gas analyzer selected from the group consisting of ultrasonic transducers, infrared spectroscopy, ultraviolet spectroscopy, gas chromatography, mass spectroscopy, mass flow meter and combinations thereof.
40. The method of claim 39, wherein the first carrier gas and the second carrier gas are selected from the group consisting of argon, nitrogen, helium and hydrogen.
41. The method of claim 40, wherein the precursor is selected from the group consisting of  $W(CO)_6$ ,  $(Me_2N)_5Ta$ ,  $(Et_2N)_5Ta$ ,  $(^1BuN)Ta(NMe_2)_3$ ,  $(^1BuN)Ta(NEt_2)_3$ ,  $TaCl_5$ ,  $TaF_5$ ,  $TiCl_4$ ,  $HfCl_4$ ,  $(Et_2N)_4Hf$  and  $XeF_2$ .